

Identification of Chlorite and Serpentine in Cosmetic or Pharmaceutical Talc

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Chlorite is the most common accessory mineral group found in high purity talc ore used in cosmetic or pharmaceutical consumer talcum products. X-ray diffraction and wet chemical analytical data obtained on geologic samples representing commercial talc ore deposits of high purity and on processed samples representing talc found in consumer talcum products indicate that clinochlore and penninite are the two chlorite minerals most commonly found in all talc samples irrespective of origin or source, but sheridanite is also found in some samples representing talc deposits that are associated with serpentinite rocks. The chlorite minerals exhibit certain well-defined X-ray diffraction characteristics which differentiate them from each other as well as from those of serpentine minerals, including the hazardous chrysotile asbestos.

Introduction

Talc as used for cosmetic, pharmaceutical and industrial purposes is probably more accurately described as a pulverized rock rather than a pulverized mineral. This is essentially because "talc" is a mixture of the mineral talc, $\text{Mg}_3\text{Si}_2\text{O}_{10}(\text{OH})_2$, and other associated minerals such as dolomite, calcite, magnesite, chlorite, tremolite, serpentine, quartz and muscovite. The amount of these associated minerals is variable and may range from merely a trace to relatively major amounts. Cosmetic talc represents material whose talc mineral content is at least 85% and preferably 90%. Pharmaceutical talc represents material of similar to slightly higher purity than cosmetic talc. Industrial talc represents relatively impure material whose talc mineral content may be as low as 30%.

Chlorite is usually the most common accessory mineral occurring in cosmetic and pharmaceutical talcs, with content ranging approximately between one and six percent. The presence of chlorite causes considerable confusion among those who perform routine examinations relating to the purity of these talcs, mainly because of the following. Chlorite is in fact a mineral group, specifically a group of phyllosilicates showing considerable isomorphous substitution between magnesium, iron, aluminum and silicon, and there are 21 different X-ray diffraction pat-

terns listed under "chlorite group" in the JCPDS file. In his study on the misidentification of asbestos in talc, Krause (1) implied that any one of the 21 chlorite minerals may be found associated with talc. Secondly, two of the most prominent X-ray diffraction peaks of chlorite (the 7 Å and the 3.5 Å peaks) correspond with the most intense serpentine peaks, and the two mineral groups may be easily mistaken for one another. A positive identification of serpentine would normally suggest the need for more detailed examination by optical and electron microscopy techniques because of the possibility that the hazardous chrysotile asbestos variety of serpentine may be present. It should be noted that the confusion relating to the identification of chlorite and serpentine in cosmetic and pharmaceutical talcs does not strictly apply to industrial talcs. In industrial talcs, chlorite occurs either in sufficient quantity to exhibit all the characteristic peaks or it does not occur at all.

The purpose of this paper is to show which of the 21 chlorites usually occur with talcs of high purity (i.e., cosmetic and pharmaceutical talcs) and to indicate some of the distinguishing physical and chemical characteristics of these chlorites. With the exception of some pertinent data in a paper by Albee (2), there is almost a total lack of published information on the nature of chlorites typically associated with talc. Knowing the types of chlorite present in talc and their distinguishing characteristics will aid in the differentiation between chlorite and serpentine and the selection of appropriate standards dur-

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ing routine X-ray diffraction work. In addition, one can estimate the chlorite content in processed talc that has been chemically analyzed, if one knows the specific chlorite mineral present in the talc (i.e., if one knows the amount of Al_2O_3 that should be assigned to the chlorite mineral).

Materials and Methods

Samples

All samples used in this study were collected by A. M. Blount and were classified into two types: (a) "geologic samples" representing talc and chlorite obtained directly from producing talc deposits in Georgia, California, Vermont, Montana, North Carolina, Alabama and Pennsylvania, and (b) "processed samples" representing talc and chlorite obtained from talc ore in Italy, Montana, North Carolina and Vermont after it was processed or prepared for use in cosmetic or pharmaceutical consumer talcum products.

X-Ray Diffraction

The X-ray diffraction patterns for measuring peak intensities were produced by using a scan speed of 2° 2θ /min and a slit width of 1° . The basal spacings of the chlorites in the processed samples were obtained by step scanning the 3.5 \AA reflection and either the quartz 1011 peak (3.34 \AA) where quartz had been added as an internal standard or the talc 006 peak (3.12 \AA) where the spacing of this peak had been previously determined for the talc deposit in question. The geologic samples were either step scanned as indicated above or scanned at $1/2^\circ$ 2θ /min through the 3.5 \AA (004) to 2.8 \AA (005) talc peaks, correcting by means of the quartz 1011 or talc 006 peaks.

Chemical Analysis

Wet chemical analyses were done on three relatively pure geologic samples of chlorite. Two of these samples were obtained from sedimentary talc deposits which are associated with carbonate (calcite and dolomite) rocks in Alabama and North Carolina. The other sample was obtained from an ultramafic talc deposit which is associated with serpentinite rocks in Pennsylvania. These three samples were selected from among two dozen chlorite samples representing over ten localities or deposits because they showed the widest range of compositional variation among the chlorites. More specifically, the latter selection was based on X-ray diffraction data and optical parameters which were obtained and evaluated for all samples under consideration. Two reference works (3,4) were used in the

evaluation of optical parameters in relation to chemical composition. The X-ray diffraction data were evaluated based primarily on the work of Brindley and others (5-7).

Results and Discussion

X-Ray Diffraction

Several researchers (2,5,6,8,9) have shown that X-ray diffraction characteristics of the chlorites, specifically the intensity and spacing of their basal reflections, depend upon their composition. For instance, the 001 (14 \AA), 003 (4.7 \AA) and 005 (2.8 \AA) basal reflections of iron-rich chlorites are relatively weak compared with the equivalent reflections of magnesium-rich chlorites. Based on this and similar observations, Brindley and other researchers (5-7) discussed procedures for determining the composition of a chlorite through the use of X-ray diffraction pattern.

Differentiation among Chlorites. Using the above information, we can determine which chlorites are present in chlorite-talc mixtures. Since the basal reflections of chlorite are the ones usually observed in mixtures of chlorite with talc, the intensity and the spacing of 001 , 002 and 004 reflections of a number of chlorites representing both geologic as well as processed samples were analyzed and plotted in Figure 1. Since measured intensities of basal reflections can be affected significantly by the degree of orientation of the layer silicate flakes, we have chosen to use the type of plot shown in Figure 1 because the value plotted is independent of the degree of orientation of the chlorite flakes (see Appendix A for further discussion). In addition, at low angles 2θ the samples is wholly within the X-ray beam, and part of the ray is lost around the sample, resulting in relatively low intensities for basal reflections. However, since this decrease in intensity relates to the slit size of the X-ray diffractometer, a correction was made for the data of Figure 1 (see Appendix B for a discussion of this correction).

Figure 1 indicates that chlorites occurring with talc, irrespective of the origin of the talc deposits, have similar diffraction characteristics. With the exception of several JCPDS chlorite samples which represent pure chlorite occurrences that are not associated with talc deposits, all samples appear close together on the plot or within the Id and If fields of Albee (2). The Id field represents a group of 21 geologic samples of chlorite found primarily in serpentinite talc deposits, and the If field represents a group of 10 geologic samples of chlorite found in carbonate talc deposits. Albee's fields are also shown in Figure 2 and discussed in the following section.

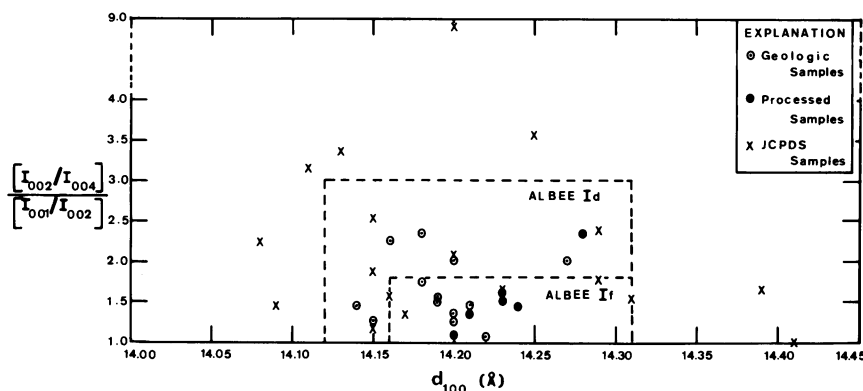


FIGURE 1. Intensity ratio and basal reflection spacings determined from x-ray diffraction data of oriented samples of chlorite found in talc deposits (geologic samples) and in processed talc (processed samples). In addition, all JCPDS chlorite samples with adequate chemical analysis information are also plotted. The *Id* and *If* fields of Albee (2) represent data from a group of 21 chlorite samples from serpentinite talc deposits and a group of 10 samples from carbonate talc deposits, respectively. See Appendix C for more detailed information on all samples plotted.

It should also be pointed out that two geologic samples and one processed sample plot relatively further away from the rest of similar samples in Figure 1 (i.e., plot above 2.0 relative intensity on the graph). This is due to two factors: experimental error and asymmetry of heavy atom distribution. The first factor is significant in the case of the processed talc which plots away from the other processed talc samples. This is because the chlorite is at the detection limit of the X-ray diffraction procedure and the peak intensity and positions were difficult to measure accurately. The two geologic samples plotting above 2.0 relative intensity have an asymmetric distribution of heavy ions which alters the intensity of 001 reflection and causes some deviation of the plotted position vertically. The problem of asymmetry in distribution of heavy ions was discussed in detail by Petruk (10).

The aforementioned conclusion that chlorites associated with talc have similar diffraction characteristics or differ very little compositionally from one region to another was also noted earlier by Berg (11) and Blount et al. (12, 13) in connection with studies of talc deposits in North Carolina, Alabama, and Montana. Furthermore, this conclusion is in agreement with chemical data (presented below) which show that of the 21 compositional types of chlorite that can possibly be associated with talc, only two or three actually do occur with talc.

Differentiation between Chlorite and Serpentine. As stated above, X-ray diffraction characteristics of chlorite occurring in talc, irrespective of origin, are generally similar. However, some apparent differences in X-ray diffraction characteristics between chlorite samples obtained from carbonate

(sedimentary) talc deposits and those obtained from serpentinite (ultramafic) talc deposits are often observed. These apparent differences are due to the fact that the chlorite samples associated with serpentinite talcs show distortions in the intensity and spacing of the 7 Å(002) and 3.5 Å(004) reflections because of the overlap of chlorite with serpentine.

In cases where chlorite comprises 6% or less of the sample and if the peaks agree in spacing and intensity to those shown in Table 1, one can with confidence identify the diffracting mineral as chlorite. Should serpentine be present in such cases, it is below the detection limit of the diffractometer since it does not cause apparent distortions in relation to the data of Table 1. Even with special treatments discussed below, serpentine will not be detected in such cases. However, in cases where distortions in intensity of *d* spacing from the characteristic pat-

Table 1. X-ray diffraction data for chlorite samples with relatively large differences in their composition.^a

<i>hkl</i>	North Carolina No. NC76.5		Pennsylvania No. T20	
	<i>I</i> ^b	<i>d</i> ^c	<i>I</i> ^b	<i>d</i> ^c
001	69	14.25	76	14.16
002	100	7.11	100	7.08
003	68	4.72	48	4.73
004	83	3.531	70	3.548
005	20	2.830	17	2.834

^a1/2° 2θ/min. scan, 1° slit; oriented sample with quartz and talc internal standards.

^b(*I*₀₀₂/*I*₀₀₄)/(*I*₀₀₁/*I*₀₀₂): North Carolina, 1.45; Pennsylvania, 1.56. Ratios corrected for loss of intensity of 001 peak (see text).

^cDetermined using basal reflections of greater than 18° 2θ (less than 5 Å). Reflections at lower 2θ angles are less accurate.

terns of Table 1 are noted, the following step scanning procedures and/or heat treatment technique can be useful in differentiating between chlorite and serpentine.

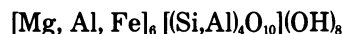
A step scan through the 7 Å or 3.5 Å region may show the presence of a double peak. The d spacing of these double peaks should be carefully measured, since it is possible for more than one chlorite to occur in processed talcs, particularly those from serpentinite associations. If one of the peaks is in the region of 3.63–3.66 Å (or 7.26–7.32 Å), heat treatment may be used as further confirmation of its identity as serpentine.

Heating a sample (oriented on a glass slide) to 500°C for three hours will yield one of two results: (a) the 7 and 3.5 Å peaks will disappear, indicating that the peaks belong to chlorite (see Fig. 2A); (b) a strip-chart scan shows that the 7 Å peak appears to have shifted to a lower 2θ value (see Fig. 2B). However, examination of a step scan taken before and after heat treatment will show that the “shifted

peak” is the serpentine peak which is visible once the chlorite peak is removed by heating. It should be noted that the heat treatment technique, although often useful, is limited by the tendency of some serpentine to show a decrease in intensity of basal reflections before the decomposition temperature of 600°C (14–16).

Chemical Analysis

The general formula for chlorite is



in which the cations are divided according to their structural position as either octahedral (Mg, Al, Fe) or tetrahedral (Si, Al) cations. This explains the exceedingly varied isomorphous substitution in the chlorite group and the fact that there are 21 different X-ray diffraction patterns listed under “chlorite group” in the JCPDS file.

In the preceding section we concluded that chlorites associated with talc deposits have similar diffraction characteristics and are, therefore, similar in composition or differ very little compositionally from one region to another. The purpose of this section is to substantiate the latter (i.e., that chlorites associated with talc have similar compositions) by evaluating compositional data on chlorites obtained here and primarily from the literature.

Table 2 contains the chemical analysis and the structural formula (based on 14 oxygen equivalents) of each of three geologic samples of chlorite. Two of these represent carbonate (sedimentary) talc deposits from Alabama and North Carolina, and the third represents a serpentinite (ultramafic) talc deposit from Pennsylvania. The samples were chosen to represent the widest range of compositional variations among the chlorites (see “Materials and Methods”). In addition, Table 2 contains analytical data obtained from the literature (11) for a fourth chlorite sample representing a carbonate talc deposit in Montana.

The data of Table 2 are plotted on the total chlorite field in Figure 3. The figure also includes the Albee *Id* and Albee *If* fields which are based on data presented by Albee (2) on two groups of chlorite: group *Id* represents 21 geologic samples of chlorite found in serpentinite (ultramafic) talc deposits or in zones bordering the serpentinite masses and referred to as “blackwall” zones, and group *If* represents 10 geologic samples of chlorite found in carbonate (sedimentary) talc deposits. On the basis of the *Id* and *If* fields as well as the other data in Figure 3, the following three points should be noted.

(1) The data on the four chlorites analyzed show that clinocllore and penninite are the only chlorites

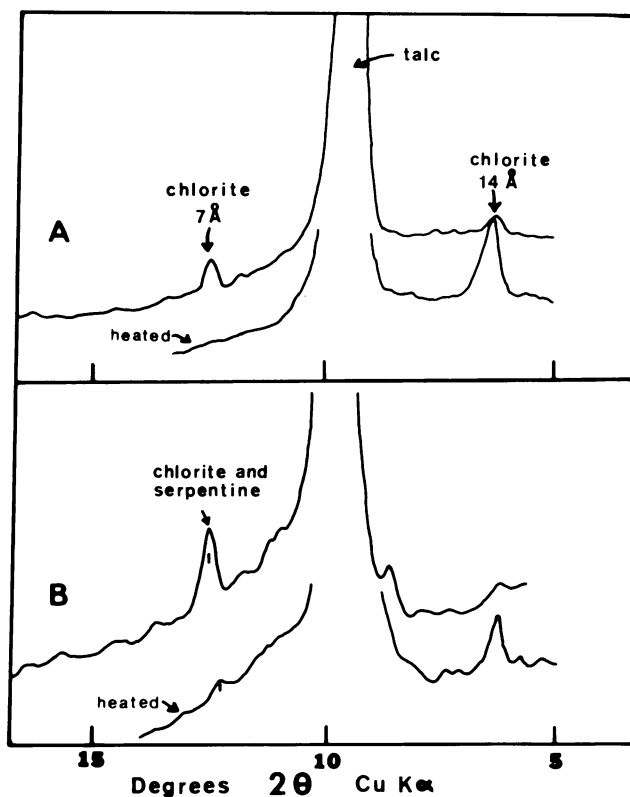


FIGURE 2. X-ray diffraction patterns illustrating the effect of heat treatment on samples of talc containing chlorite and serpentine: (A) the 7Å peak disappears with heat treatment, indicating that the peak belongs to chlorite; (B) the A peak remains but with a slight shift in d position after heat treatment, indicating that serpentine is present along with chlorite.

Table 2. Chemistry of chlorite samples associated with talc deposits.

	SiO ₂ , %	Al ₂ O ₃ , %	Fe ₂ O ₃ , %	FeO, %	MgO, %	CaO, %	H ₂ O ⁺ , %	H ₂ O ⁻ , %	Formula (based on 14 oxygen equivalents)
Montana No. 3316-6 ^b	32.41	18.16	0.61	6.16	31.24	0.04	12.73	0.31	Al _{1.05} Fe ³⁺ _{0.04} Fe ²⁺ _{0.48} Mg _{1.37} Al _{0.95} Si _{3.05} O ₁₀ (OH) ₈
Alabama No. LN6	32.01	16.42	0.92	2.28	31.59	0.91	12.55	1.89	Al _{0.97} Fe ³⁺ _{0.07} Fe ²⁺ _{0.19} Ca _{0.09} Mg _{1.6} Al _{0.89} Si _{3.11} O ₁₀ (OH) ₈
North Carolina No. NC76.5	33.73	17.42	0.85	4.01	27.63	1.79	11.35	1.87	Al _{1.2} Fe ³⁺ _{0.03} Fe ²⁺ _{0.32} Ca _{0.18} Mg _{3.98} Al _{0.77} Si _{3.24} O ₁₀ (OH) ₈
Pennsylvania No. T20	28.50	20.95	1.01	11.62	24.33	0.72	10.31	1.42	Al _{1.24} Fe ³⁺ _{0.07} Fe ²⁺ _{0.96} Ca _{0.07} Mg _{3.60} Al _{1.19} Si _{2.81} O ₁₀ (OH) ₈

^aThe Montana, Alabama, and North Carolina samples are associated with carbonate (sedimentary) talc deposits; the Pennsylvania sample with a serpentinite (ultrabasic) talc deposit.

^bChemical data from Berg (11). In addition to CaO, 0.01% of Na₂O and K₂O are also included in Berg's analysis. However, he excluded all three from the formula as probable impurities.

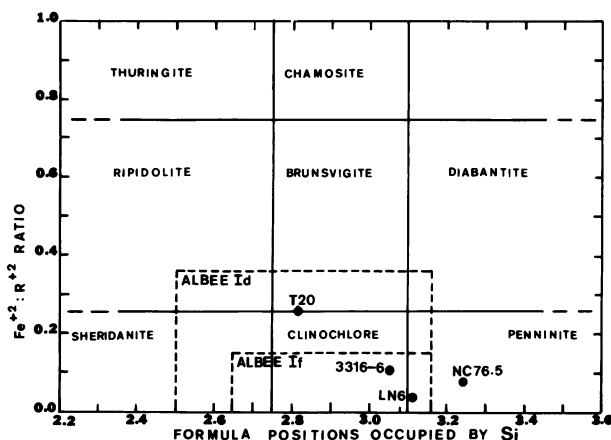


FIGURE 3. Plot of compositions of analyzed chlorite on Foster's (17) classification scheme. Samples 3316-6, LN6 and NC76.5 are from carbonate talc deposits, and sample T20 is from a serpentinite talc deposit (see Table 2). The *Id* and *If* fields of Albee (2) represent data from two groups of 31 chlorite samples (see Fig. 1).

associated with both serpentinite (ultramafic) as well as carbonate (sedimentary) talc deposits. In addition, the *Id* and *If* fields suggest sheridanite as another type of chlorite found in these deposits.

(2) The higher iron contents of group *Id* suggest the presence of more types of chlorites (e.g. ripidolite, brunsvigite, diabantite) in talc deposits; however, this is not actually the case since the higher iron contents seem to belong only to the "blackwall" chlorites which occur outside the talc-bearing zones and generally do not appear in processed talcs. This is verified by a detailed study of the Vermont serpentinite talc deposits (18) which shows a very rapid increase in the iron content of chlorites once they occur outside the talc zone.

(3) The fact that the chlorite sample NC 76.5 (which represents a sedimentary carbonate talc from North Carolina) plots outside the *If* field of

Albee (which represents the sedimentary carbonate talc group), may be explained as follows. Albee does not indicate the silica content of the chlorite samples in his graphs and tables on which the data for fields *Id* and *If* were based; thus the simplifying assumption was made by the writers that half the aluminum was octahedrally coordinated and the other tetrahedrally. If this assumption were made for the North Carolina chlorite (NC 76.5), it would fall inside Albee's *If* field.

Summary and Conclusions

Geologic chlorite samples, representing pure or relatively pure chlorite obtained directly from talc deposits, and processed chlorite samples, representing chlorite obtained from processed cosmetic-grade talc, are quite similar in terms of their X-ray diffraction characteristics and, therefore, their chemical composition, irrespective of the origin of the talc deposits (i.e., sedimentary "carbonate" talc deposits or ultrabasic "serpentinite" talc deposits). Some apparent differences in X-ray diffraction characteristics between chlorite samples obtained from carbonate talcs and those obtained from serpentinite talcs are due to the fact that the latter samples show distortions in the intensity and spacing of the 7 Å (002) and 3.5 Å (004) peaks because of the overlap of chlorite with serpentine.

In the X-ray identification of chlorite in talc, the JCPDS patterns most applicable are those of clinochlore (19-749 or 12-242), penninite (10-183) and sheridanite (7-77). However, some serpentinite talcs may contain more than one chlorite mineral, and these chlorite minerals may vary slightly in Al content and *d* spacing, thus producing a double peak which may become apparent during step scanning. Nevertheless, the intensities are not affected, since the intensity ratios are very similar for all chlorites which occur with talc.

Appendix A: Degree of Orientation of the Chlorite Flakes

The basal intensities of chlorite (or any other phyllosilicate) can vary significantly due to the degree to which the flakes in the X-ray sample have aligned themselves parallel to one another. In order to eliminate the influence of this factor, we plotted intensities as the ratio $[I_{002}/I_{004}] / [I_{001}/I_{002}]$ which is independent of the degree of orientation of the flakes as shown below:

The basic formulas for intensity are discussed by Schoen (19) for both "random" as well as "oriented" samples. For randomly oriented flakes,

$$I = K(j) \frac{1}{V^2} (A\theta) |F|_2 \left(\frac{1 + \cos^2 2\theta}{\sin \theta \sin 2\theta} \right)$$

and for oriented flakes,

$$I = K(j) \frac{1}{V^2} (A\theta) |F|_2 \left(\frac{1 + \cos^2 2\theta}{\sin 2\theta} \right)$$

where I = the integrated intensity of the diffraction peak, K is a constant which depends upon certain physical constants and the geometry of the diffractometer, j is the multiplicity factor, V is the volume of the unit cell, $A\theta$ is the absorption factor, F is the structure factor, and θ the Bragg diffraction angle. Thus, for any reflection, the oriented and random intensities differ by a factor of $1/\sin \theta$. The ratio used in this report $[I_{002}/I_{004}]$ will differ by the following factor for random as compared with oriented:

$$\frac{[(1/\sin \theta_{002}) / (1/\sin \theta_{004})]}{[(1/\sin \theta_{001}) / (1/\sin \theta_{002})]}$$

but $\lambda = 2d_{hkl} \sin \theta$.

Therefore,

$$\sin \theta = \lambda/2d_{hkl}$$

which means that the samples differ by a factor of:

$$[d_{002}/d_{004}]/[d_{001}/d_{002}] = 1.0$$

Thus, the ratio $[I_{002}/I_{004}]/[I_{001}/I_{002}]$ is independent of the degree of orientation of the particles in the samples.

Table A-1

Classification	Origin	Ratio	Spacing, Å
Processed talc	Italy	1.64	14.23
	"	1.51	14.23
	Montana	1.34	14.21
	"	2.35	14.28
	N. Carolina	1.09	14.20
	Vermont	1.45	14.24
	Georgia	2.02	14.27
	California	1.26	14.15
	Vermont	2.27	14.16
	"	2.02	14.20
Geologic samples	"	1.09	14.22
	Montana	1.26	14.20
	"	2.35	14.18
	"	1.44	14.21
	N. Carolina	1.51	14.19
	"	1.34	14.20
	"	1.45	14.14
	Alabama	1.76	14.18
	Pennsylvania	1.56	14.19
		1.63	14.23
JCPDS # 7-77	sheridanite		
7-165	grocharite (sheridanite)	1.33	14.17
12-242	leuchtenbergite (clinochlore)	1.16	14.15
19-749	clinochlore	1.76	14.29
7-78	leptochlorite	1.85	14.15
7-171	diabantite	1.56	14.16
16-362	chlorite Ib (diabantite)	2.38	14.29
7-76	ripidolite	2.52	14.15
12-243	aphrosiderite	2.22	14.08
13-29	thuringite	3.13	14.11
21-1227	thuringite	1.42	14.09
22-712	nimite	8.89	14.20
12-185	kotschubeite	3.54	14.25
7-160	kotschibeite	1.52	14.31
7-166	daphnite	3.33	14.13
10-183	penninite	1.66	14.39
16-362	chlorite Ia	2.08	14.20
20-671	kammererite	1.00	14.41

Appendix B: Divergence Slit Size of the X-Ray Diffractometer

Schoen (19) discussed the effect of the divergence slit size on basal intensities. For instance, at low angles of θ , part of the X-ray beam may be lost, resulting in relatively low basal intensities; however, the decrease in intensity relates to the slit size of the X-ray diffractometer according to the formula:

$$L = \left(\frac{\gamma}{57.3} \right) \left(\frac{R}{\sin \theta} \right)$$

where L is the length of the sample surface in centimeters, γ is the angular aperture of the divergence slit in degrees, R is the goniometer radius in centimeters, and θ is the angle between the X-ray beam and the sample.

According to the above formula, the length of the sample should be 5.48 cm if, as in this study, one uses a 1° slit size and a goniometer radius of 17 cm for the characteristic basal reflection (001) of chlorite at $6.2^\circ 2\theta$. However, since the length of the sample surface actually used in this study is 4.6 cm, the intensity of the basal (001) reflection was corrected by a factor of 1.2.

It should be noted that if one uses a 3° divergence slit size, 72% of the impinging X-ray is lost. This loss may cause the 001 peak of chlorite to be completely absent, especially in samples that contain a relatively small amount of chlorite. With the absence of the characteristic 001 chlorite peak, one can easily mistake chlorite for serpentine when, in fact, chlorite is present and serpentine is not.

Appendix C: X-Ray Data for Samples Plotted in Fig. 1

Intensity ratio, $[(I_{002}/I_{004})]/[(I_{001}/I_{002})]$, and basal reflection spacings (d_{100} in angstrom units) as well as place of origin information on all chlorite samples plotted in Fig. 1 are given in Table A-1.

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